

WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau

<p>(51) International Patent Classification 6: <b>D21C 9/147</b></p>	<p><b>A1</b></p>	<p>(11) International Publication Number: <b>WO 97/15715</b></p> <p>(43) International Publication Date: <b>1 May 1997 (01.05.97)</b></p>
<p>(21) International Application Number: <b>PCT/SE96/01154</b></p> <p>(32) International Filing Date: <b>18 September 1996 (18.09.96)</b></p> <p>(30) Priority Data: <b>9503720-6</b>      <b>23 October 1995 (23.10.95)</b>      <b>SE</b></p> <p>(71) Applicant: <b>SUNDS DEFIBRATOR INDUSTRIES AB</b> <b>[SE/SE]; S-851 94 Sundsvall (SE).</b></p> <p>(72) Inventors: <b>BOKSTRÖM, Monica; Bjärne 3251, S-860 25</b> <b>Kovland (SE). MELLANDER, Pär; Bråkagatan 4 A, 2tr,</b> <b>S-856 30 Sundsvall (SE). NORDÉN, Solveig; Odalvägen</b> <b>14, S-862 41 Njurunda (SE).</b></p> <p>(74) Agent: <b>SUNDQVIST, Hans; Sunds Defibrator Industries AB,</b> <b>Strandbergsgatan 61, S-112 51 Stockholm (SE).</b></p>		<p>(83) Designated States: <b>AU, BR, CA, CN, JP, NO, NZ, RU,</b> <b>European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB,</b> <b>GR, IE, IT, LU, MC, NL, PT, SE).</b></p> <p><b>Published</b> <i>With international search report.</i></p>
<p>(54) Title: <b>OXYGEN DELIGNIFICATION OF LIGNOCELLULOSIC PULP IN TWO STEPS</b></p>		
<p>(57) Abstract</p> <p>A method of oxygen delignification of pulp from lignocellulosic material at medium concentration in two steps. An extended delignification is obtained in that the temperature in the first step is held below 90 °C, that the difference in temperature between the steps is lower than 20 °C. The pressure in the first step is 4-10 bar and in the second step 2-5 bar, and the pressure in the first step is higher than in the second step. The oxygen addition to the first step is high, 25-50 kg/ton pulp, that alkali is added only to the first step for obtaining high alkalinity in the pulp. 25-50 kg alkali per ton pulp. The stay-time in the first step is 10-30 min and in the second step 45-180 min.</p>		

10000

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AM	Armenia	GB	United Kingdom	MY	Malawi
AT	Austria	GE	Georgia	MX	Mexico
AU	Australia	GN	Ghana	NE	Niger
BD	Burkina Faso	GR	Greece	NL	Netherlands
BE	Belgium	HU	Hungary	NO	Norway
BF	Burkina Faso	IE	Ireland	NZ	New Zealand
BG	Bulgaria	IT	Italy	PL	Poland
BJ	Benin	JP	Japan	PT	Portugal
BR	Brazil	KE	Kenya	RO	Romania
BY	Belarus	KG	Kyrgyzstan	RU	Russian Federation
CA	Canada	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KZ	Republic of Korea	SE	Sweden
CG	Congo	LA	Laos	SG	Singapore
CH	Switzerland	LJ	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovakia
CM	Cameroon	LR	Liberia	SN	Senegal
CN	China	LT	Lithuania	SS	Swaziland
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	LV	Latvia	TG	Togo
DE	Germany	MC	Monaco	TJ	Tajikistan
DK	Denmark	MD	Republic of Moldova	TT	Trinidad and Tobago
EE	Estonia	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	UG	Uganda
FI	Finland	MN	Mongolia	US	United States of America
FR	France	MR	Mauritania	UZ	Uzbekistan
GA	Gabon			VN	Viet Nam

WO 97/15715

1

PCT/SE96/01154

## OXYGEN DELIGNIFICATION OF LIGNOCELLULOSIC PULP IN TWO STEPS

This invention relates to a method of oxygen delignification of ligno-cellulosic material at medium concentration, i.e. 8-16%.

Since the introduction of oxygen delignification at medium pulp concentration not much development work has been devoted to this process. Since chlorine free bleaching and the closing of bleach plants have become a matter of current interest, extended delignification, i.e. a further lowering of the kappa number, by means of oxygen has increasingly become more interesting. Extended delignification by oxygen in one or several steps, however, can result in deteriorated pulp quality. Right conditions, however, can yield several advantages.

It should be possible to maintain the yield of the pulp better than at extended cooking, i.e. cooking to lower kappa number.

At a multi-step method it should be possible to distribute the chemicals between the steps in order to obtain optimum conditions in every step. Even other conditions could then be optimized.

The present invention relates to a method of extended oxygen delignification so that a lower kappa number is obtained without thereby deteriorating the properties of the pulp. By extended delignification according to the invention, the total delignification can amount to 50-70% of the lignin content (kappa number) of unbleached pulp. The method is carried out at medium pulp concentration in two subsequent steps. The characterizing features of the invention are apparent from the attached claims.

The invention is described in greater detail in the following with reference to the accompanying Figure illustrating schematically an installation for carrying out the method according to the invention.

At the installation shown, digested pulp at medium concentration, i.e. 8-16%, is pumped by a first pump 1 from brown pulp washing to oxygen delignification. A first mixer 2 is used for admixing oxygen

WO 97/15715

2

PCT/SE96/01154

and alkali to the pulp. The pulp is thereafter fed into a first reactor 3, in which the first delignification is carried out. The pulp is directed from there, possibly by a second pump 4, via a second mixer 5 for admixing steam and possibly additional oxygen to a second reactor 6 for the second delignification step. After the second reactor 6 the pulp is fed to a blow tank 7 and from there to subsequent processing steps.

The method, thus, implies that the delignification is carried out in two subsequent steps. In the first mixer 2 both a high alkali addition and a high oxygen addition are made. This implies a charge of 25-50 kg alkali (NaOH) per ton pulp, preferably 25-35 kg/ton. This necessary alkali charge possibly can partially be obtained by a carry-over from the brown pulp washing. The charge in the mixer 2 then can be reduced in a corresponding degree. The oxygen charge shall be 25-50 kg/ton pulp, preferably 30-40 kg/t.

The temperature of the pulp at the feed into the reactor 3 shall be below 90°C, preferably 75-90°C. This implies that the reaction in the reactor 3 can be carried out at the temperature of the pulp when it comes from the brown pulp washing. The staytime in reactor 3 shall be relatively short, 10-30 min, preferably 15-25 min.

The pressure in the first reactor 3 shall be 4-10 bar. The high pressure, combined with the high alkalinity of the pulp and the high oxygen charge, results in a high delignification speed. At the same time, the speed for the cellulose degradation is held on a low level, due to the relatively low temperature and short staytime.

After the first delignification step in the first reactor 3 the pulp is fed to the second delignification step in the second reactor 6. The temperature in the second reactor 6 shall be above 90°C, i.e. higher than in the first reactor 3. The difference in temperature

WO 97/15715

3

PCT/SE96/01154

however, shall be less than 20°C, preferably 10-15°C. For bringing about the required increase in temperature, steam is supplied to the second mixer 5.

The pressure in the second reactor 6 shall be 2-5 bar and lower than in the first reactor 3. The staytime should be relatively long, 45-180 min, preferably 60-120 min.

The second delignification step foremost is a long extraction step where in relation to the first step the increased temperature and the extended staytime yield extended delignification. At temperatures above 90°C, thus, good extraction/leaching speed is obtained.

Due to the fact that no additional alkali is charged in the second step, not even for compensating for the consumption in the first step, the alkalinity of the pulp can be held relatively low in the second step. Hereby substantially cellulose degradation is avoided, in spite of high temperature and long staytime.

In the second mixer 5 possibly a small amount of oxygen can be added, which can be up to 5 kg/ton pulp. The oxygen charge in the first step can thereby be completed in order to increase the partial pressure of the oxygen.

The staytime in the second step is determined in relation to the temperature, in order to achieve optimum results, i.e. intended extended delignification without deterioration of the pulp properties. Higher temperature, thus, means shorter staytime.

The invention, of course, is not restricted to the embodiment shown, but can be varied within the scope of the invention idea.

WO 97/15715

4

PCIT/SE96/01154

# Claims

1. A method of oxygen delignification of pulp from lignocellulosic material at medium concentration in two steps, characterized in that an extended delignification is obtained in that the temperature in the first step is held below 90°C and in the second step above 90°C, that the difference in temperature between the steps is lower than 20°C, that the pressure in the first step is 4-10 bar and in the second step 2-5 bar, that the pressure in the first step is higher than in the second step, that the oxygen addition to the first step is high, 25-50 kg/ton pulp, that alkali is added only to the first step for obtaining a high alkalinity in the pulp, 25-50 kg alkali per ton pulp, and that the staytime in the first step is 10-30 min and in the second step 45-180 min.

2. A method as defined in claim 1, characterized in that the temperature increase between the two oxygen steps is 10-15°C.

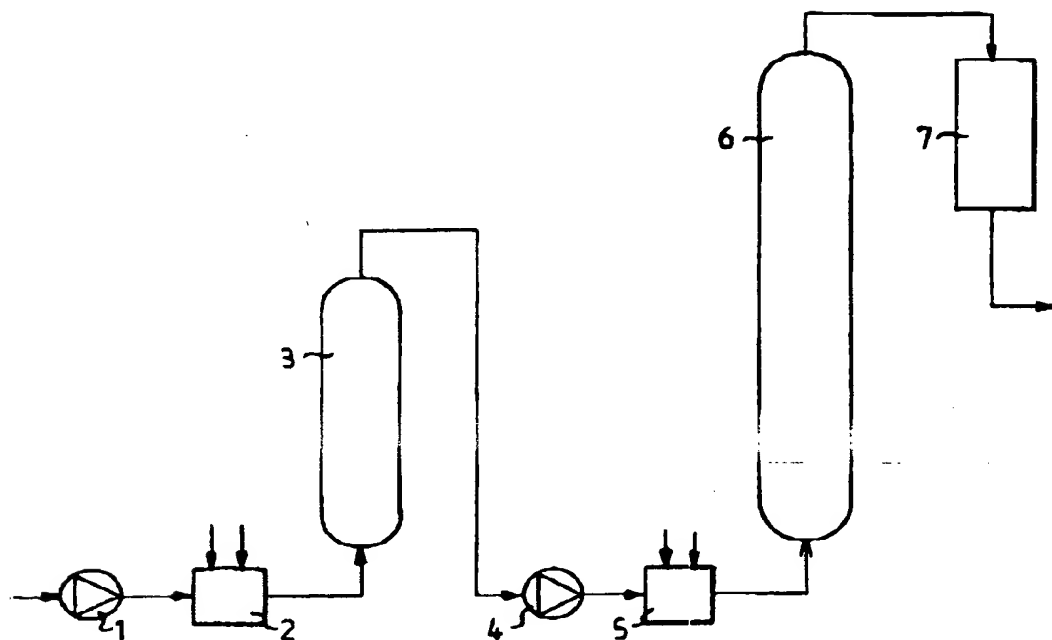
3. A method as defined in claim 1, characterized in that the staytime of the pulp in the first step is 15-25 min and in the second step 60-120 min.

4. A method as defined in any one of the preceding claims, characterized in that additional oxygen is charged in an amount of 0-5 kg/ton pulp to the second step.

WO 97/15715

1/1

PCT/SE96/01154



1

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 96/01154

## A. CLASSIFICATION OF SUBJECT MATTER

IPC6: D21C 9/147

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: D21C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5217575 A (ÅKE BACKLUND), 8 June 1993 (08.06.93), column 2, line 67 - column 4, line 5	1-4
A	US 4946556 A (J. ROBERT PROUGH), 7 August 1990 (07.08.90), column 1, line 45 - line 56; column 2, line 61 - column 3, line 29	1-4

☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

## \* Special categories of cited documents:

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubt on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

\*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

\*X\* document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

\*Y\* document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

\*Δ\* documents member of the same patent family

Date of the actual completion of the international search

Date of mailing of the international search report

16 December 1996

09-01-1997

Name and mailing address of the ISA/  
Swedish Patent Office  
Box 5055, S-102 42 STOCKHOLM  
Facsimile No. +46 8 666 02 86

Authorized officer  
Marianne Bratsberg  
Telephone No. +46 8 782 25 00



**INTERNATIONAL SEARCH REPORT**  
Information on patent family members

28/10/96

International application No.

PCT/SE 96/01154

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A- 5217575	08/06/93	CA-A- 1337843 FI-B,C- 91896 JP-A- 2145883 SE-B,C- 467582 SE-A- 8803705	02/01/96 13/05/94 05/06/90 10/08/92 19/04/90
US-A- 4946556	07/08/90	FR-A,B- 2646446 JP-A- 3014686 SE-C- 503759 SE-A- 9001187	02/11/90 23/01/91 26/08/96 26/10/90